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STRAIN AGING EFFECTS IN TUNGSTEN DUE TO CARBON

by Joseph R. Stephens and G. Willy Form

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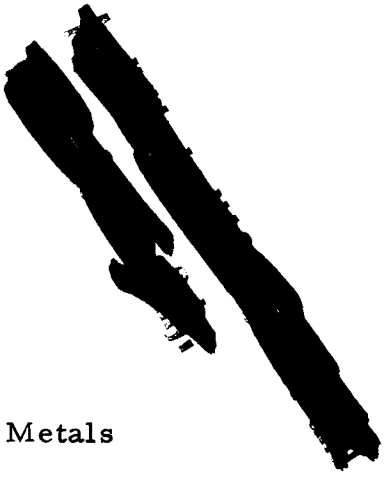

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STRAIN AGING EFFECT IN TUNGSTEN DUE TO CARBON

by Joseph R. Stephens[1] and G. Willy Form[2]

LEWIS RESEARCH CENTER

NGS-35213

ABSTRACT

A

Strain aging characteristics of tungsten containing 8 and 40 ppm carbon were determined. Completely recrystallized tensile specimens were prestrained various amounts at 700° F and aged at 1200°, 1500°, and 1800° F for times up to 4 hours.

Results show that dislocation locking by solute atoms was weak for the lower carbon level as evidenced by the lack of a yield drop in either the prestrained or aged condition. Specimens containing 40 ppm carbon exhibited a yield drop during the prestrain test. Aging for 120 minutes at 1200° F did not result in a return of the yield drop. The strain aging parameter, which reflects a summary effect involving recovery, strain aging, and precipitation hardening, was negative for short aging times but increased to a positive value after aging for longer times. Aging at 1500° and 1800° F resulted in a return of the yield drop, and the strain aging parameter was positive for all aging times. Explanation of these phenomena are proposed in terms of recent theories.

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INTRODUCTION

As the need for higher melting point materials has increased, extensive research into the mechanical properties of the refractory metals

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columbium, molybdenum, tantalum, and tungsten has been conducted. Since each of these metals has a body-centered cubic structure, it is to be expected that the sharp yield point and strain aging phenomena resulting from an interaction between solute atoms and dislocations would occur in these metals in a manner analogous to that observed in steels [1-4]. This has been shown to be true for columbium, molybdenum, and tantalum [5] where the sharp yield point and strain aging have been attributed to various interstitial solute atoms present in these metals. The sharp yield point and strain aging have also been reported for tungsten; however, to date, insufficient data are available to determine which interstitial impurities produce these phenomena in tungsten. Two independent investigators have suggested that hydrogen may produce discontinuous yielding and strain aging in tungsten. The first of these [6] observed a definite yield point in tungsten single crystals after annealing in hydrogen at 600° C (1112° F) for 90 minutes. The second investigator [7] attributed the rapid hardening rate of prestrained specimens annealed at temperatures up to 600° C (1112° F) to the diffusion of hydrogen to unpinned dislocations. In both of these investigations an analysis of the hydrogen content of the tungsten was not available, and there was insufficient data to substantiate the authors' suggestions.

In a more recent investigation [8] a yield drop was observed in tungsten containing small quantities of carbon. This suggested that strain aging in tungsten should occur in a manner analogous to that observed in other refractory metals and other body-centered cubic metals containing small amounts of interstitial solute elements. The present investigation was undertaken to verify this aspect by studying

the kinetics of strain aging in tungsten containing various amount of carbon.

EXPERIMENTAL PROCEDURE

Material

Commercial tungsten rods were received in the as-swaged condition having a polished ground surface. The tungsten rods, 1/4 inch in diameter and approximately 7 feet in length, were prepared from the same powder batch in an attempt to achieve uniform purity throughout all the test specimens. The major impurities contained in the rods are listed in table I.

Specimen Preparation

Button heat tensile specimens having a 0.140-inch-gage diameter and 1.00-inch-gage length were centerless ground from the 1/4-inch-diameter rods. The tensile specimens were annealed at 3700° F for 2 hours at a pressure of less than 1×10^{-5} millimeter of mercury. Commercial tungsten rod of this diameter, that is, this amount of cold work, normally has a recrystallization temperature of approximately 2600° F. However, to add carbon to the tungsten specimen a diffusion anneal of 3500° F was required [8]. In order to realize a uniform grain size throughout both the specimens to which carbon was added and those to be tested having the original carbon content, a recrystallization temperature above the diffusion anneal temperature was desirable. The resulting grain size from the recrystallization treatment was approximately 0.064 millimeter in diameter as determined by a circle-intercept method.

The tensile specimens were ground to a 0.010 inch oversize diameter. The final diameter (0.130 inch) of the specimens to which carbon was added was achieved by a two-step process. Prior to surface carburization, 0.005-inch was removed from the diameter of the specimens by electropolishing in order to achieve a clean, uniform surface. It was observed that this procedure enhanced the adherency of the carbon on the surface of the specimens during carburization. After carburization and subsequent homogenization, the carbon-rich layer remaining on the surface of the tensile specimens was removed by again electropolishing 0.005 inches from the diameter of the specimens. The reduction in the transition temperature resulting from electropolishing the surface [9] allowed testing of the specimens at a temperature appreciably below the lowest aging temperature so that further aging during test was unlikely to occur. Specimens to be tested having the original carbon content were electropolished directly to the 0.130 inch gage diameter.

To study the effect of carbon concentration on strain aging of tungsten one series of specimens was tested with the original carbon level of 8 ppm which is generally near the minimum carbon content in commercial tungsten. A second series of specimens with a carbon content of 40 ppm was selected based on the previous investigation [8] which indicated that strain aging might be expected to occur as evidenced by the yield drop in specimens with 36 to 60 ppm carbon. The addition of carbon was accomplished by heating the specimens in a propane atmosphere and subsequently giving them a high-temperature homogenization anneal as described previously [8].

Chemical analyses and microstructural studies revealed a uniform concentration of carbon across the cross section of the test specimens after annealing them for 4 hours at 3500° in vacuum and subsequently removing the remaining carbide layer by electropolishing.

Based on the previously observed transition temperature of 695° F for specimens with 36 ppm carbon [8] a test temperature of 700° F was selected for this investigation. The temperature of the specimen was determined by means of a Pt-Pt 13 percent Rh thermocouple in contact with the end of the specimen. The specimens were tested in vacuum (<5 microns) in an Instron Tensile Machine at a constant cross head speed of 0.05 inch per minute. A series of specimens were loaded in the tensile machine to calibrate the cross head movement with the amount of strain in the specimen. After unloading, the reduced diameter and extended length were determined. The prestrain was expressed in terms of the plastic strain within the specimen by the equation

$$\epsilon = \ln(L/L_o)$$

where

ϵ natural strain

L_o original length

L length after prestraining

The maximum amount of uniform elongation that normally occurs in tungsten specimens tested at this temperature (700° F) is approximately 15 to 20 percent. Therefore, a maximum prestrain of 15 was selected. Specimens with 8 ppm carbon were prestrained 0.03, 0.10, and 0.15, while those with 40 ppm carbon were prestrained 0.04, 0.075, 0.10, and 0.15.

Aging

The specimens were aged in vacuum at a pressure of 5×10^{-4} millimeter of mercury. After the furnace was brought to the desired temperature for aging, the specimens were lowered into the heated zone. Aprroximately 3 minutes were required for the specimens to reach the aging temperature. After aging for the desired time, the specimens were removed from the heated zone and allowed to cool to room temperature. Aging temperatures selected were 1200°, 1500°, and 1800° F, and times up to 240 minutes were used at each aging temperature.

Method of Evaluation

The criterion used to measure strain aging can best be explained with the use of figure 1. The first curve depicts the initial prestrain test and subsequent unloading assuming that the yield point is present. After aging, reloading of the specimen is illustrated in the second or third curves. The strain aging parameter ΔYS selected is the difference between the load (P) at the lower yield point during reloading or the load at 0.2 percent offset if smooth yielding occurs, point r, and the load prior to unloading in the prestrain test, point p, divided by the cross sectional area A_p of point p, that is,

$$\Delta YS = \frac{P_r - P_p}{A_p}$$

Metallographic Studies

For the metallographic examination of the prestrained and aged state, the specimens were electroetched in a 2 percent sodium hydroxide solution ro reveal the substructure by means of etch pits [10].

EXPERIMENTAL RESULTS

Strain Aging in Tungsten - 8 ppm Carbon

The sharp yield was not observed in any of the specimens containing 8 ppm carbon during the initial prestrain tests nor upon subsequent aging and reloading. The time dependence of the strain aging parameter is shown in figures 2 and 3 for the two extreme aging temperatures investigated (1200° and 1800° F, respectively). It was observed for several specimens that for zero aging time, that is, immediate reloading after prestraining, yielding occurred at the same load as the load prior to unloading in the prestrain test. Therefore, the curves for each prestrain are extrapolated to a strain aging parameter equal to zero for zero aging time. It should be noted in each of these figures that the strain aging parameter ΔYS is negative (as determined in fig. 1, curves 1 and 3) for all aging conditions, that is, the yield stress of the aged specimen is less than the flow stress prior to unloading of the prestrained material. Also characteristic of each of the figures is that for all three prestrains aging for 30 and 60 minutes resulted in a decrease of the strain aging parameter with increasing aging time. Furthermore, for each prestrain, the curves for the strain aging parameter first reached a minimum, then a maximum, and subsequently dropped again (with the exception of the curve for the 0.03 prestrained specimens aged at 1800° F) as the aging time was increased at both aging temperatures.

Strain Aging in Tungsten - 40 ppm Carbon

A sharp yield was observed for all the specimens containing 40 ppm carbon during the initial prestrain tests. The time dependence of the aging parameter is plotted in figures 4 to 6 for the three aging tem-

peratures. It should be noted in figure 4 that for 30- and 60-minute aging periods at 1200° F, the strain aging parameter ΔYS is negative, that is, the yield stress upon reloading is less than the flow stress during initial prestraining. Also of interest in figure 4 is that after aging for 30 minutes the curves for the higher prestrains (0.10 and 0.15) exhibited the lowest minimum.

By increasing the aging temperature to 1500° F the change in yield stress was positive for all aging times as shown in figure 5. It should be noted that each curve for the lower three prestrains (0.04, 0.075, and 0.10) passes through a maximum while ΔYS for the 0.15 prestrained specimens increases with aging times up to 120 minutes.

After aging at 1800° F, ΔYS reaches a maximum at a very short time of aging, 30 minutes or less (fig. 6), for all prestrains. Continued aging at this temperature results in a gradual decrease in the magnitude of the strain aging parameter, suggesting that the specimens have over aged.

Activation Energy for Yield Point Return in Tungsten Containing 40 Carbon

After aging at 1200° F for times up to 120 minutes, a sharp yield point was not obtained upon reloading of the specimens. From the time necessary to obtain a return of the yield point upon aging at various temperatures an estimate of the activation energy for the strain aging process in the tungsten containing carbon can be made. Specimens strained 0.04 were aged at 1200° F for time intervals exceeding 120 minutes to obtain the return of a sharp yield point. Specimens were also aged at 1500° F for times less than 30 minutes to repress the occurence

of a sharp yield point. Figure 7 is a plot of $\ln(1/t)$ (where t is the aging time) against $1/T$ (where T is the aging temperature in degrees absolute). The straight line in figure 7 is drawn through those points which indicated the times at which the sharp yield point would first be observed at those temperatures in question. From the slope of this line, the activation energy was calculated to be 50.4 kilocalories per mole.

Microstructural Studies

Tungsten - 8 ppm carbon. - Figure 8 illustrates the change in microstructure of a specimen prestrained 0.04 and then aged at 1200° F. Figure 8(a) shows the recrystallized microstructure. After straining to 0.04 strain (fig. 8(b)), very little change in dislocation density is observed as evidenced by the number of etch pits. (The etching technique used produces etch pits that have been shown to correspond to dislocations intersecting the surface [10]). No apparent change in dislocation density (etch pit density) was observed after the aging treatments of 30 and 120 minutes as shown in figures 8(c) and (d), respectively. The photomicrographs were taken on the electropolished and etched surface on one tensile specimen so that the sequence of events could be followed within the same specimen. Therefore, due to the radius of curvature of the specimen, the photomicrographs may not be in focus at the edges. Figure 9 illustrates the same sequence of events for a specimen prestrained 0.15 and aged at 1200° F. A comparison of the recrystallized specimen (fig. 9(b) reveals that a large change in dislocation density is evident from the density of etch pits present. Figure 10 shows the substructure of the 0.15 prestrained

specimen after aging for 120 minutes at 1200° F. It appears that the etch pits are aligned on the slip planes of the grain shown in this figure.

Tungsten - 40 ppm Carbon. - The same sequence of events appeared to occur for both carbon levels upon prestraining and aging the specimens at 1200° F. Also the microstructures of tungsten of both carbon levels after aging at 1800° F bear a strong similarity to each other. Figures 11 and 12 reveal the resulting microstructures of the specimens containing 40 ppm carbon prestrained and then aged at 1800° F. The arrows in figure 11 point to carbides precipitated at the grain boundaries. In figure 12(b), etch pits in the two grains indicated by arrows seem to be aligned on slip planes of each particular grain. Neighboring grains do not exhibit the same density of etch pits due to different orientations [10]. Upon aging, rows of etch pits perpendicular to the slip planes appear as shown in figures 12(c) and (d). At a higher magnification (fig. 13), a rectangular network of etch pits resulting from aging at 1800° F for 120 minutes is more easily observed.

DISCUSSION OF RESULTS

Strain Aging in Tungsten - 8 ppm Carbon

The lack of a yield point in the prestrain tests or after aging and subsequent testing indicates that pinning of dislocations was weak in tungsten containing 8 ppm carbon. Therefore, all of the results must be attributed to recovery upon annealing in this temperature range of 1200° to 1800° F. Experimental evidence from studies of the recovery process in heavily worked tungsten [11] illustrated that

recovery as determined by resistivity measurements takes place at a temperature as low as 750° F by removal of vacancies and that polygonization resulted as determined by an etch-pit technique after aging at 1475° F for 30 minutes [12]. In metals having a high stacking fault energy, which is believed to be the case for tungsten due to the separation of the recovery and recrystallization temperatures, both cross slip and climb are taking place during recovery.

As noted previously the curves in figures 2 and 3 (1200° and 1800° F aging temperatures) exhibit a minimum after aging for 60 minutes. Upon extending the aging time to 120 minutes a maximum in ΔYS is obtained for both aging temperatures. The behavior suggests that for short aging times the initial recovery process annihilation of dislocations (and consequent softening) as a result of the formation of attractive junctions that result in a reduction of dislocation line length [13]. Annihilation of dislocations can also occur as a result of intersections of dislocations of opposite sign. At some time in the recovery process, a metastable network of dislocations forms. This formation of subgrains or polygonization will tend to strengthen the specimens as has been shown previously for tungsten [12]. Therefore, the increase in ΔYS in the curves between 60 and 120 minutes at both aging temperatures seems to be characteristic of a change in the recovery process and may be attributed to the formation of subgrains. Continued aging for longer times allows growth of the network to occur with an accompanying renewed softening. Alternately, the increase in ΔYS between 60 and 120 minutes could be attributed to weak dislocation locking by the carbon atoms. Due to an insufficient

amount of carbon, however, the locking was not strong enough to completely overcome the softening due to recovery.

The larger decrease in ΔYS for the higher prestrains, which is particularly evident upon aging at 1800° F (fig. 3), can be attributed to the increased driving force for recovery, that is, the increase in strain energy for the higher prestrains.

Finally, of interest in figures 2 and 3 is the temperature dependence of the change in ΔYS after short aging times (30 and 60 min). Aging the 0.10 prestrained specimens for 60 minutes at 1200° F resulted in ΔYS = -2600 pounds per square inch, while at 1800° F ΔYS = -4930 pounds per square inch for the same amount of prestrain and time of aging. For the 0.15 prestrained specimens, ΔYS had values of -3185 pounds per square inch and -7700 pounds per square inch after aging for 60 minutes at 1200° and 1800° F, respectively. This approximately twofold change in ΔYS for the two aging temperatures suggests that this temperature dependency of the initial stage of recovery may reflect a change in mechanism. Cross slip is believed to occur at lower recovery temperatures and play a limited part in recovery, while at higher temperatures climb occurs and is believed to be the more important process [13]. The change in mechanism of recovery from cross slip to climb upon increasing the aging temperature from 1200° to 1800° F could then account for the twofold change in ΔYS. These processes would be expected to be more pronounced with increasing prestrain, thus for only 0.03 percent the dislocation density was probably not increased a sufficient amount (fig. 8) to result in large amounts of recovery occurring.

Strain Aging in Tungsten - 40 ppm Carbon

Yield point phenomena. - The two most generally accepted theories to explain the yield point in b.c.c. metals are the "dislocation locking theory" as proposed by Cottrell and Bilby [1] whereby solute atoms pin the dislocations until, under an applied stress, the dislocations break away from their solute atmosphere with the resulting increase in number of mobile dislocations leading to the observed yield point, and the "dislocation multiplication theory" proposed by Johnston and Gilman [14] and expanded to the b.c.c. metals by Hahn [15] who assumed that the anchored dislocations remain locked and the yield point is a consequence of rapid multiplication of mobile dislocations and the stress dependence of dislocation velocity. Which one of these theories is accepted does not seem to be of consequence to this particular investigation. The primary point of interest lies in the fact that pinning of dislocations is necessary before the yield point is observed.

In this investigation, it was observed that 8 ppm carbon is not sufficient to cause the yield point in tungsten, while 40 ppm is sufficient. Cottrell and Bilby [1] proposed that 1 or 2 carbon atoms per site along a dislocation line would be sufficient to lock the dislocations. Based on this idea, 8 ppm carbon should pin the dislocations in the annealed specimens if a dislocation density of approximately 1×10^8 centimeters per cubic centimeter is assumed. As discussed recently by Clark [16], however, if it is assumed that the number of interstitial atoms per dislocation site may be as high as 100 for pinning to occur, then 8 ppm carbon would probably not be a sufficient amount to pin the dislocations present in the annealed specimens, but

40 ppm would be expected to lock the dislocations and thus produce the yield point.

1200° F Aging temperature. - The time dependence of the strain aging parameter shown in figure 4 for the 1200° F aging temperature can be explained as follows: for short times of aging (30 and 60 min) the negative change in the strain aging parameter, and the lack of sharp yielding upon reloading of the aged specimens indicates that an effective interaction between solute atoms and dislocations has not occurred. Therefore, recovery again appears to be the predominant factor effecting the mechanical properties after aging at low temperatures for short periods of time. A similar observation has been observed in molybdenum [17] where at strains greater than 5 percent recovery effects tended to predominate over the effects of strain aging, thus overall weakening occurred.

For longer aging times ΔYS again increased and also reached a positive value that is in contrast for the tungsten specimens containing 8 ppm carbon where ΔYS was always negative for similar aging conditions (fig. 2). Based on the previous discussion, two explanations could possibly account for this behavior in the tungsten - 40 ppm carbon.

First, polygonization occurs after extended aging as evidenced by the rectangular network of dislocations shown in figure 13, which will lead to strengthening as noted previously. Secondly, after longer aging times, the carbon atoms may have diffused toward the dislocations tending to block their motion but not interacting sufficiently or in large enough number for an observable yield point. The combination of

these two processes and the fact that more carbon was available thus leading to stronger pinning of the dislocations may account for the greater increase in ΔYS past the minimum in the curves for the 40 ppm carbon specimens as compared with the 8 ppm carbon series of specimens.

1500° F Aging temperature. - Increasing the aging temperature to 1500° F resulted in sharp yielding of all the specimens upon reloading. This suggests that the shortest times of aging employed were sufficient to allow the carbon atoms to diffuse to the dislocations and thus lock them. As shown in figure 5, the locking strength (as indicated by ΔYS) exhibits a maximum as a function of aging time. The fact that maximum locking occurs at longer times with increasing prestrain is believed due to the facts that dislocation density increases with increasing prestrain and that sufficient carbon is not present to effectively lock all the dislocations. Extended aging time leads to annihilation of some of the dislocations until a density is reached where maximum locking occurs. The decrease in ΔYS after reaching a maximum value for the three lower prestrains (fig. 5) is typical for age hardenable alloys when overaging occurs, supporting the view [4] that strain aging leads to actual precipitation along the dislocation line during the aging treatment. The higher values of ΔYS for the 0.15 prestrained specimens aged for 90 and 120 minutes is believed to be due to the increase in dislocation density, which provides more sites for nucleation of precipitates and hence increased strengthening results from the finer dispersed carbides. Apparently aging for 120 minutes at 1500° F was not long enough for overaging to occur for the 0.15 prestrained specimens.

1800° F Aging temperature. - The time dependence of ΔYS shown in figure 6 indicates that overaging occurs after very short periods of time for all prestrains investigated. This suggests that diffusion of carbon at this temperature is sufficiently fast for precipitates to form very rapidly.

Activation Energy For Yield Point Return

It is expected that the return of the yield point is dependent upon the rate of diffusion of the carbon atoms to the free dislocations produced during prestraining. The value of 50.4 kilocalories per mole calculated from figure 7 is in good agreement with the reported values of 59.0 [18] and 50.6 [19] kilocalories per mole for the diffusion of carbon in tungsten but lower than other values (100 and 110 kcal/mole [20]) also appearing in the literature. However, the activation energy calculated in this investigation may be associated with strain-induced diffusion for which the activation energy is thought to be lower than that for lattice diffusion. Also of interest concerning the value of 50.4 kilocalories per mole for the activation energy for the yield point return is that it is much higher than the value of 10 to 15 kilocalories per mole reported elsewhere for strain aging in tungsten [7] where hydrogen was postulated to be the diffusing interstitial impurity and would be expected to have a lower activation energy.

Microstructural Studies

The change in dislocation arrays upon straining (50 percent reduction in area) and aging of tungsten has been studied previously by several investigators [12, 21], and it was clearly established that

recovery takes place in the temperature range used here for the aging treatments. However, the well-defined network of subgrains illustrated in the previous studies were not observed in the present investigation. Instead, rectangular arrays of dislocations were delineated as shown in figures 10 and 13 where the etch pits appear to be lined up along slip lines. Similar observations have previously been made on molybdenum [22]. By comparing figures 11 and 12, it can be seen that, after similar aging treatments, the dislocation density remains much greater in the heavily deformed specimen (fig. 12). As previously discussed, this would lead to more sites of easy nucleation for precipitates.

CONCLUSIONS

The following significant conclusions can be drawn from the results presented in this study on strain aging of tungsten:

(1) A carbon level of 8 ppm is not sufficient to produce a yield drop under all prestrains and aging conditions investigated.

(2) With a carbon level of 40 ppm, a yield drop is observed in all prestrain tests. The yield point return is observed after short aging times (15 min) at 1500° and 1800° F, whereas at 1200° F the yield point return is not observed until after 480 minutes of aging.

(3) The yield point parameter ΔYS, which reflects a summary effect involving recovery, strain aging, and age hardening, is negative or positive, depending upon the relative contribution of recovery and carbon-dislocation interaction.

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TABLE I. - MAJOR IMPURITIES IN TUNGSTEN

TENSILE SPECIMENS

Element	Al	Cr	Fe	Ni	Si	Ti	V	Mo	O	N	H	C*
Composition, ppm	0.8	1.7	4.3	0.6	4.1	0.1	0.5	35	4	1	4	8

*Carbon content increased to 40 ppm in one series of specimens for strain-aging study.

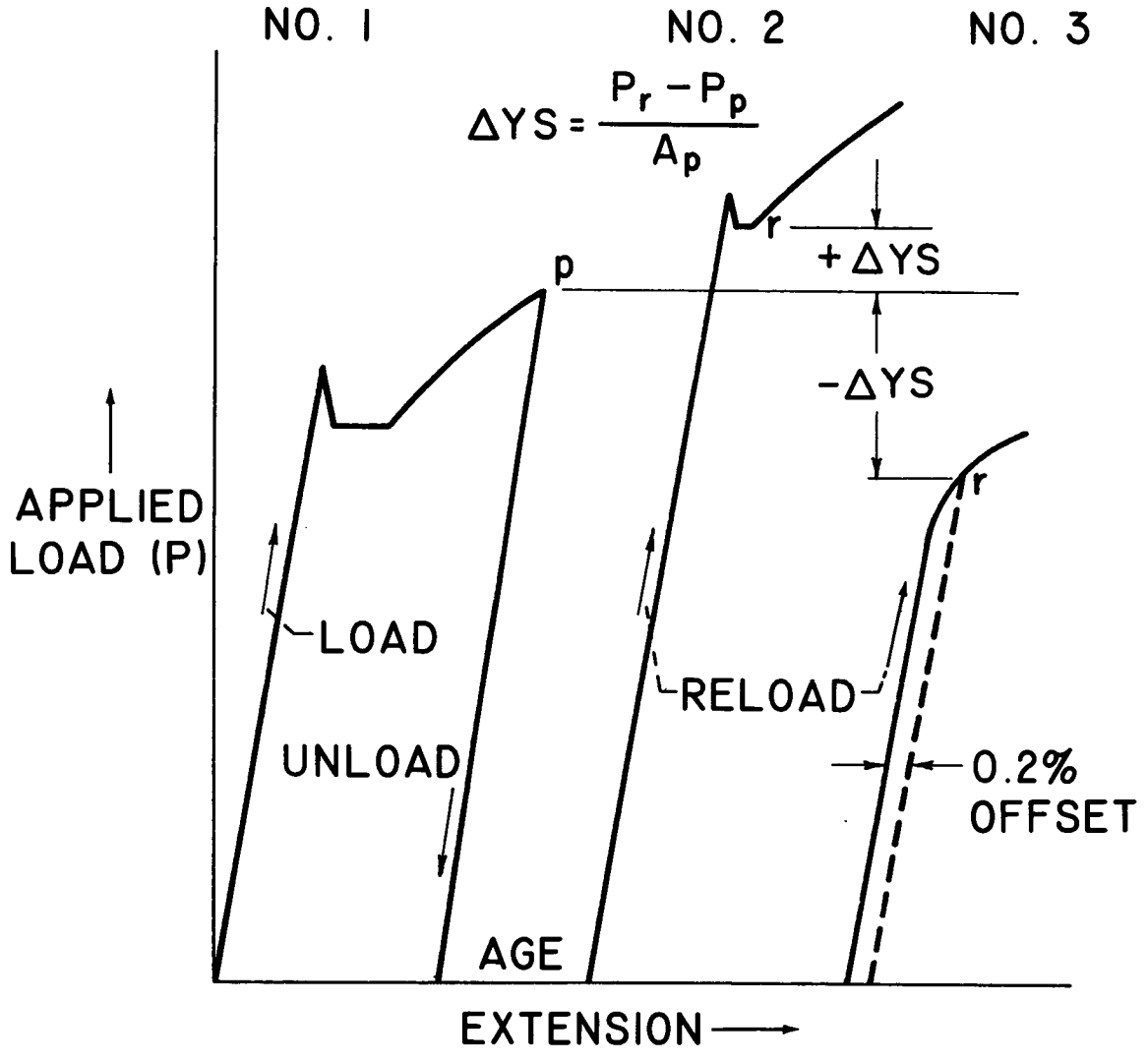

Figure 1. - Determination of strain aging parameter.

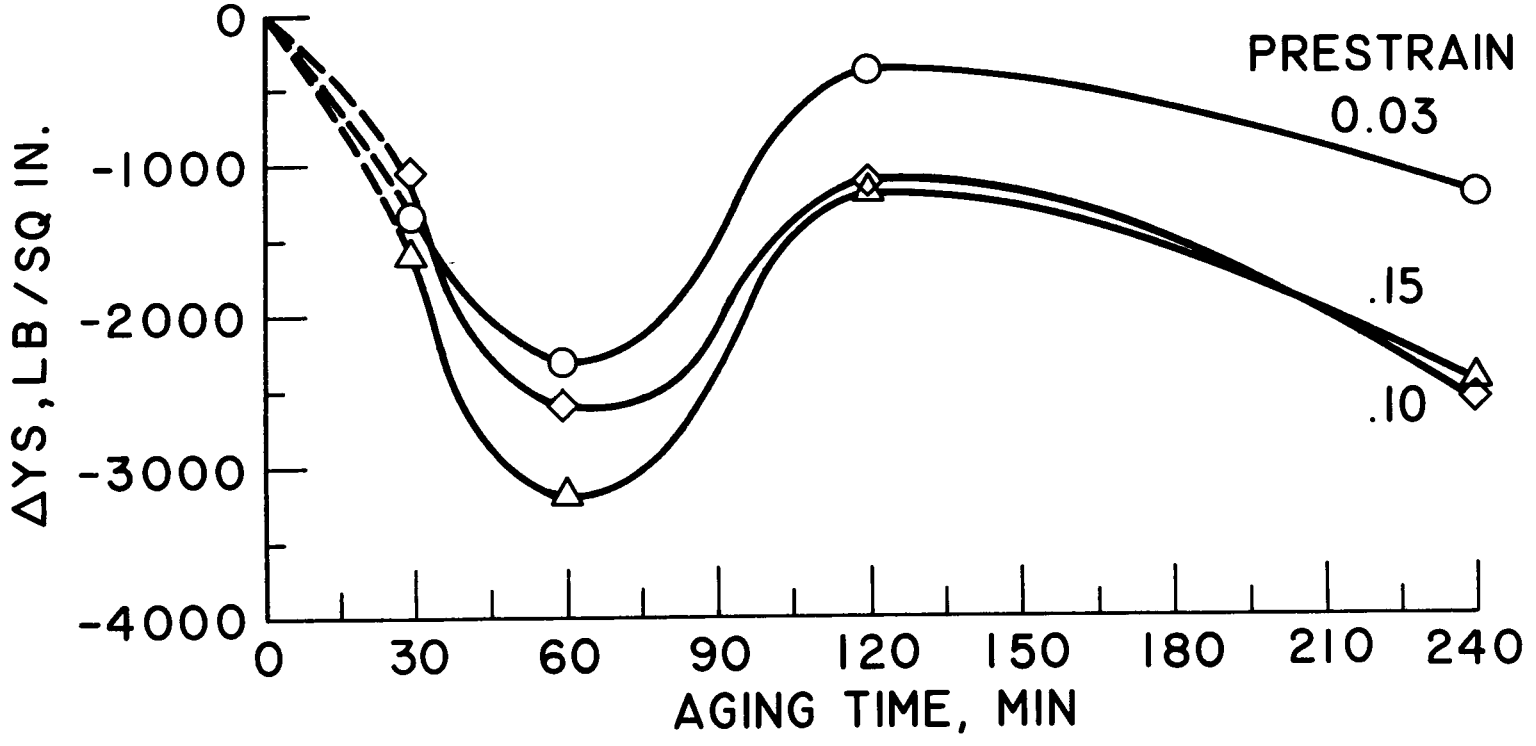

Figure 2. - Time dependence of strain aging in tungsten. Carbon content, 8 ppm; aging temperature, 1200° F.

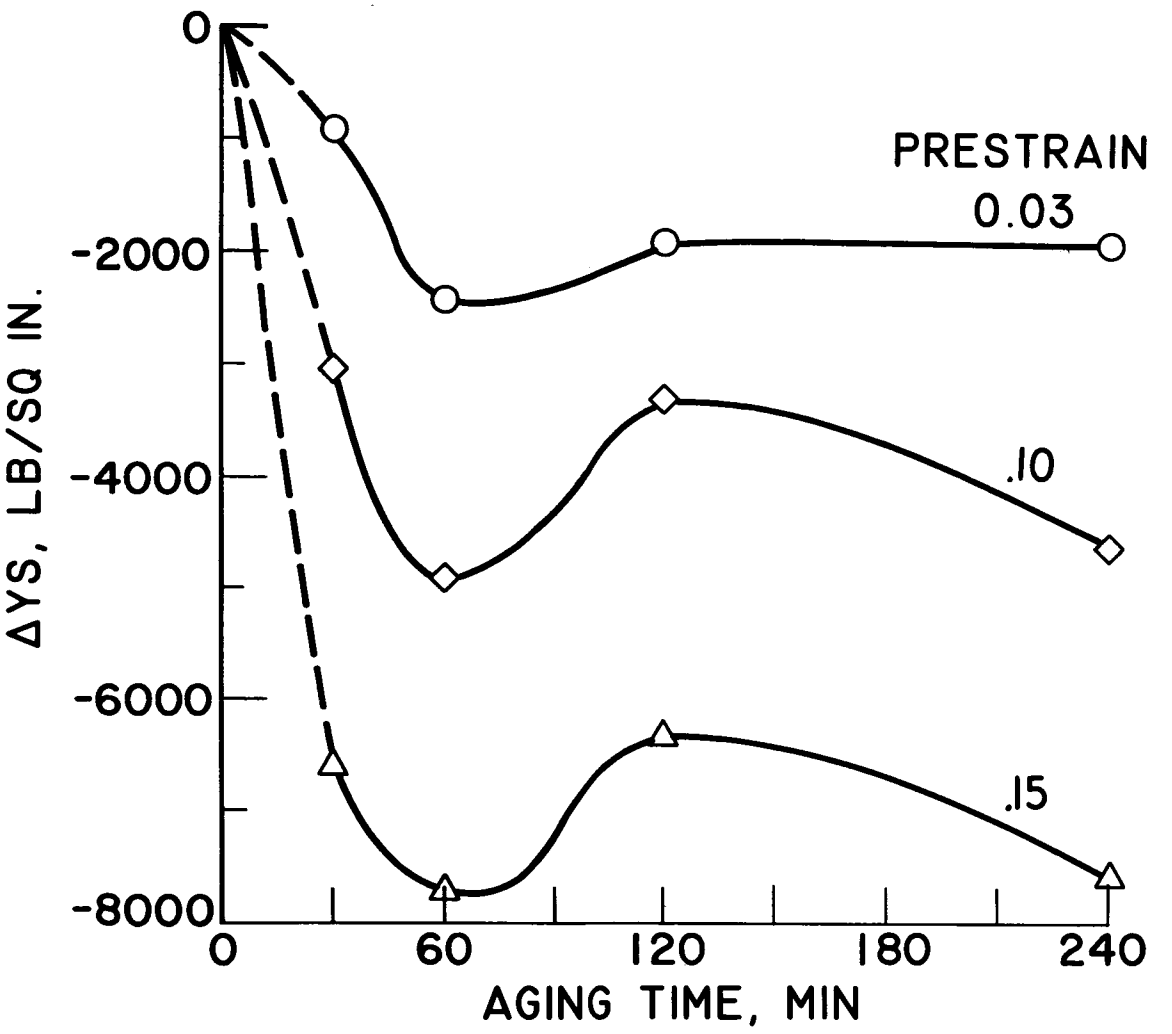

Figure 3. - Time dependence of strain aging in tungsten. Carbon content, 8 ppm; aging temperature, 1800° F.

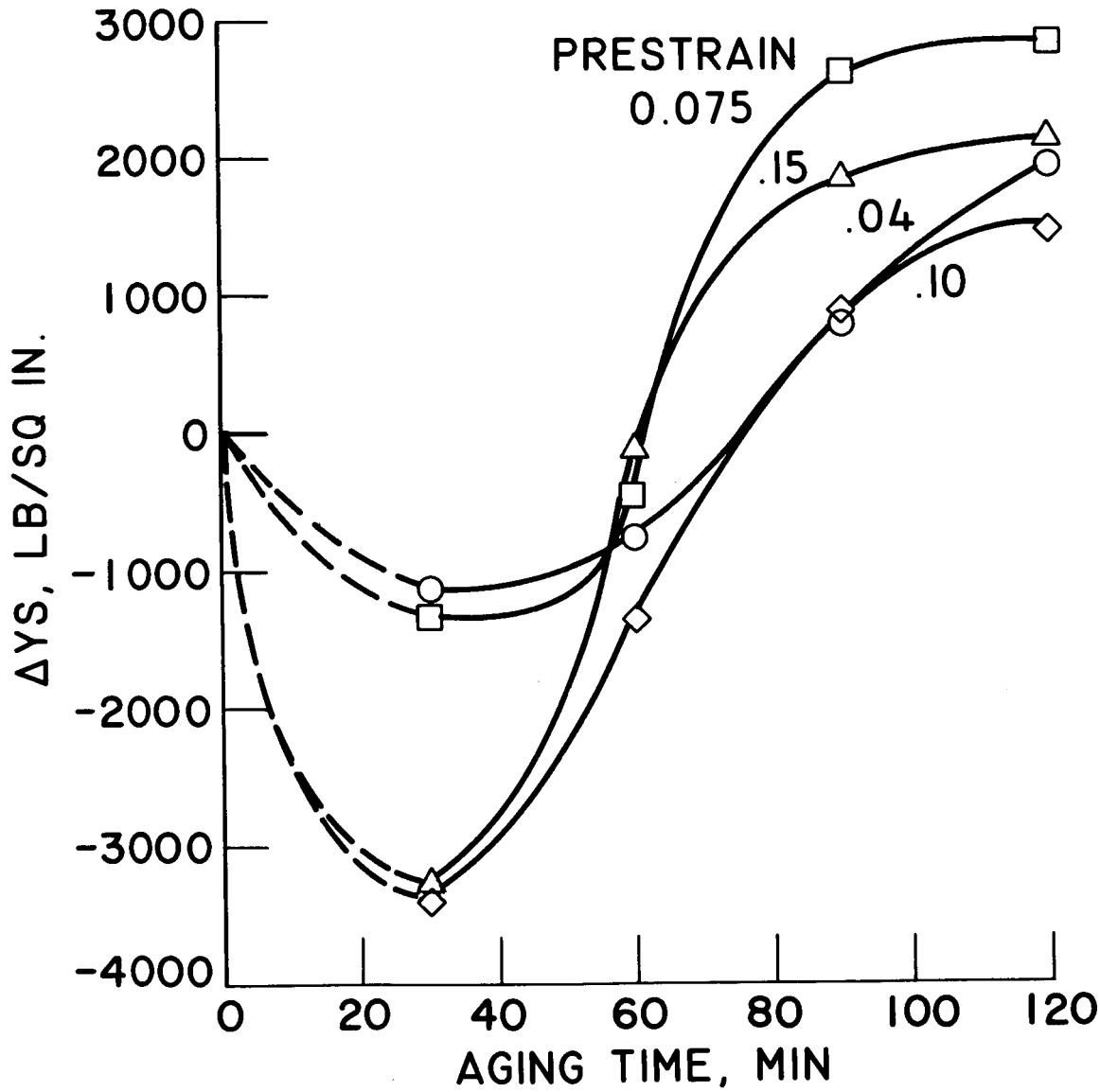

Figure 4. - Time dependence of strain aging in tungsten. Carbon content, 40 ppm; aging temperature, 1200° F.

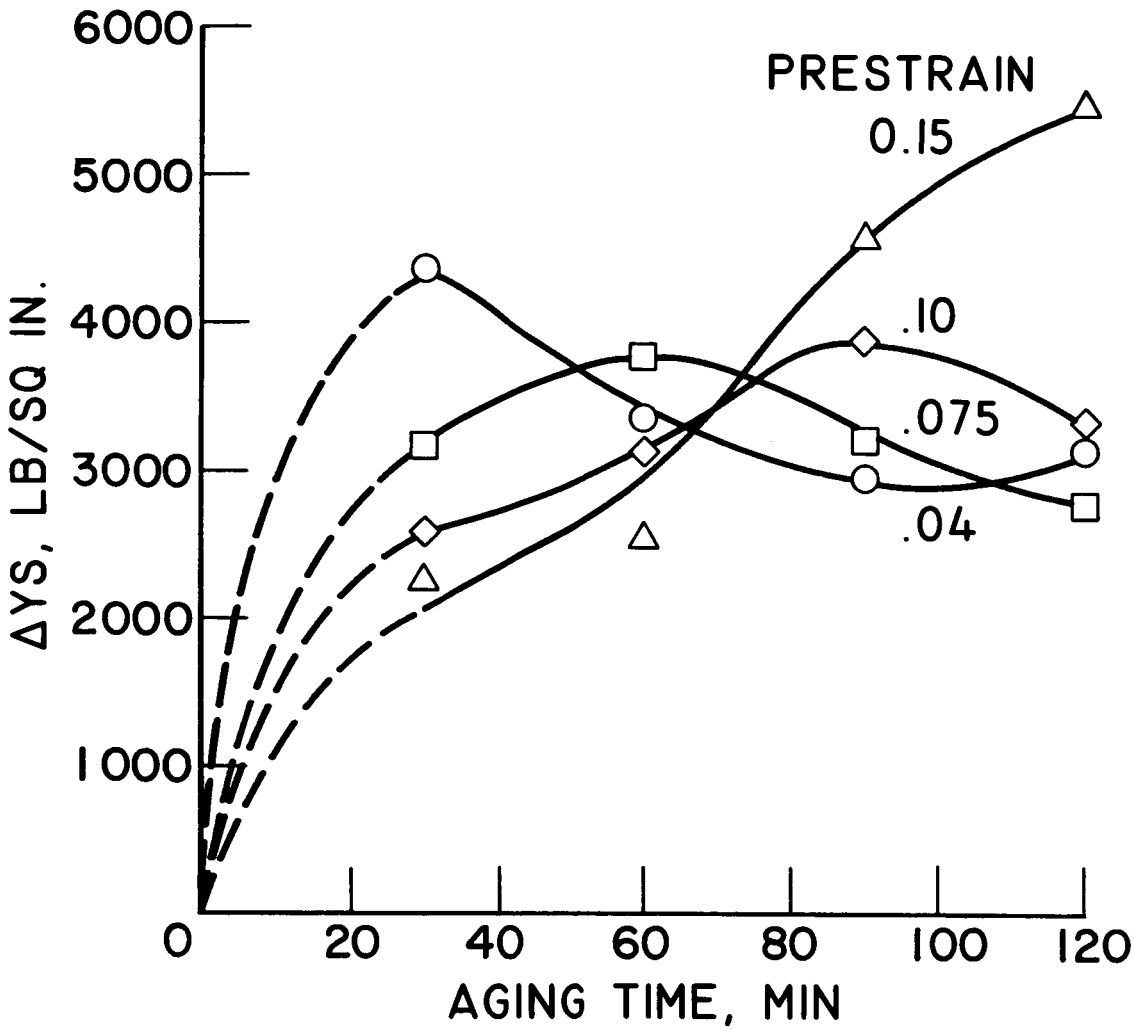

Figure 5. - Time dependence of strain aging in tungsten. Carbon content, 40 ppm; aging temperature, 1500° F.

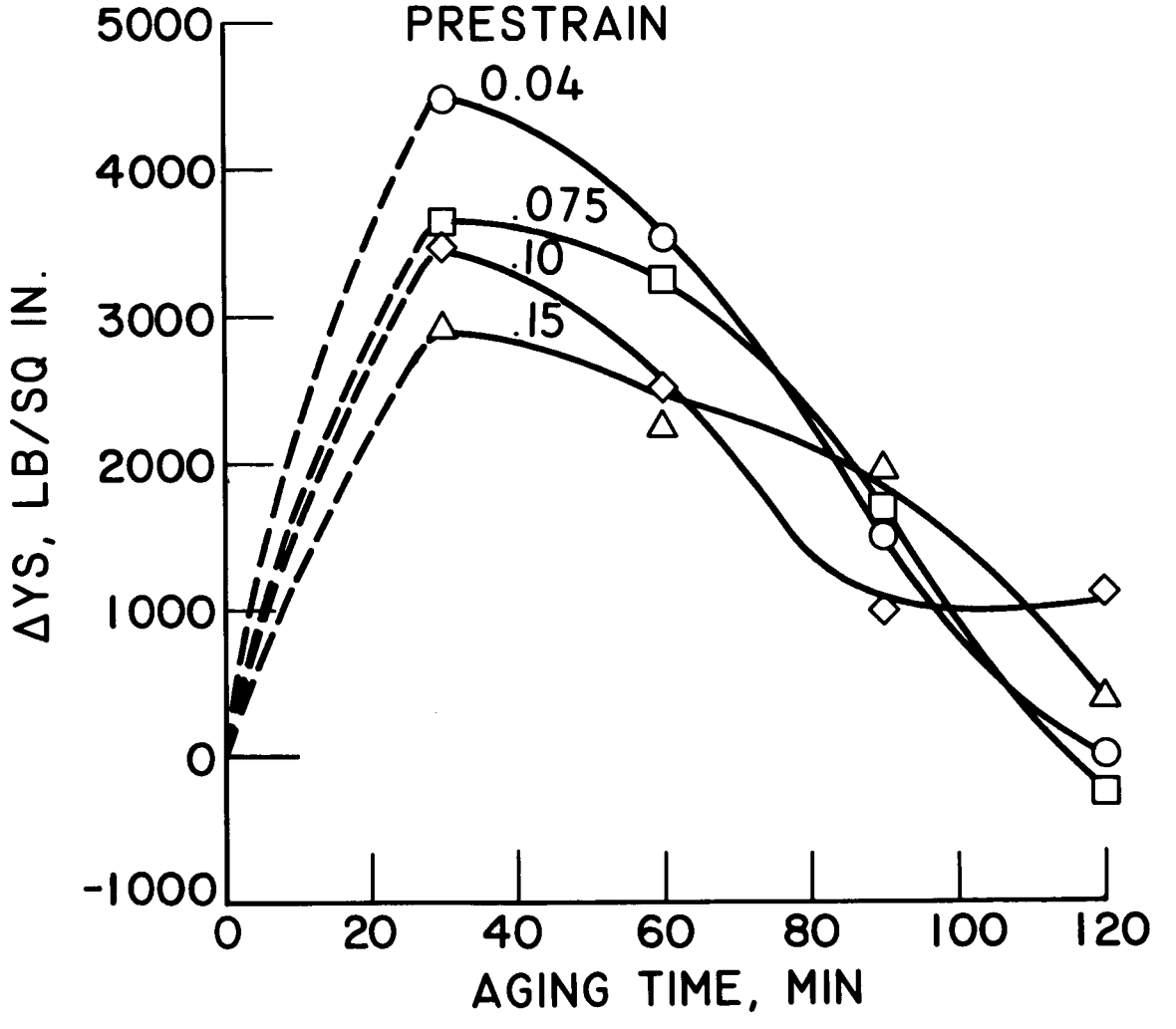

Figure 6. - Time dependence of strain aging in tungsten. Carbon content, 40 ppm; aging temperature, 1800° F.

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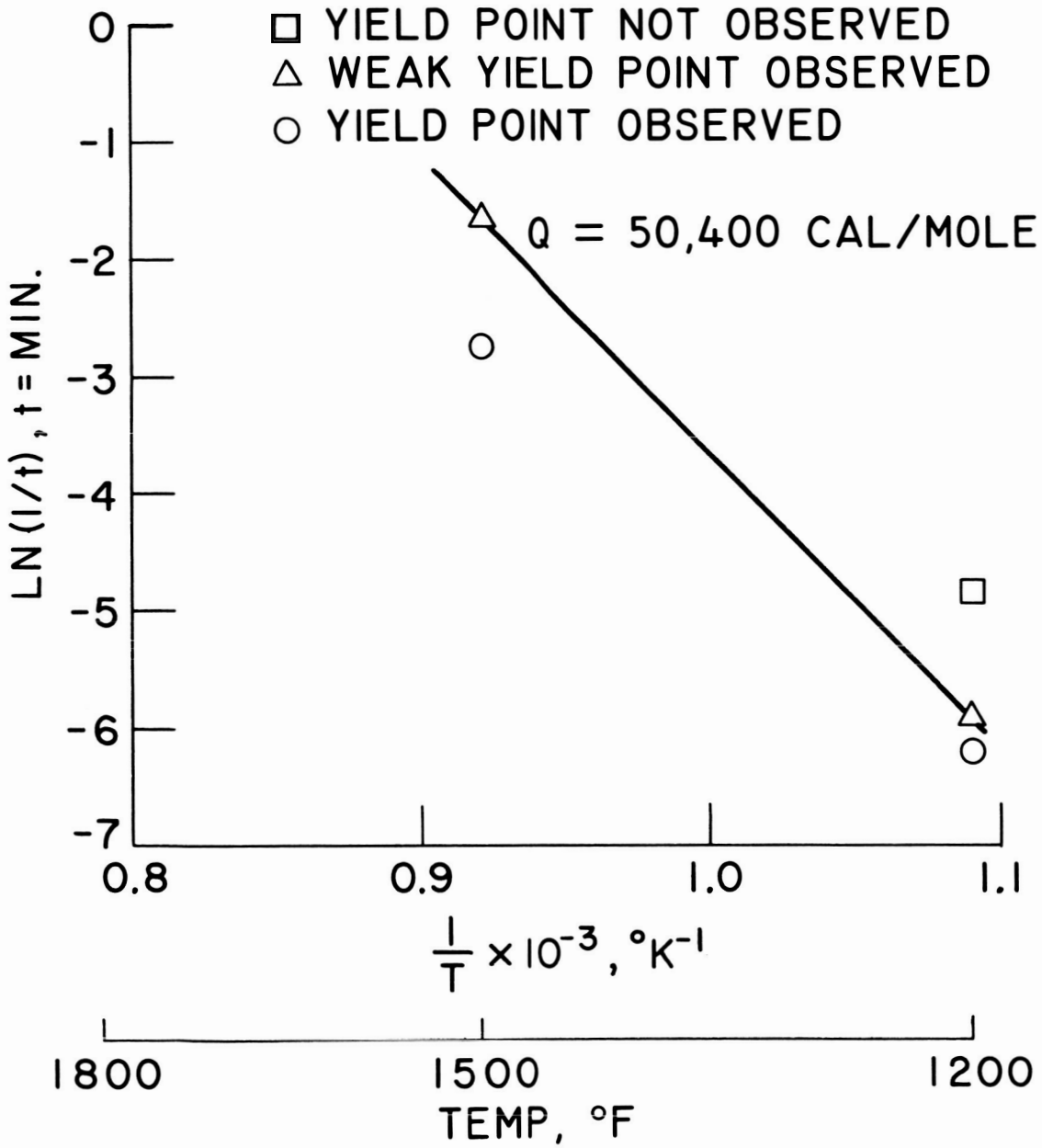

Figure 7. - Activation energy for the return of yield point in tungsten.

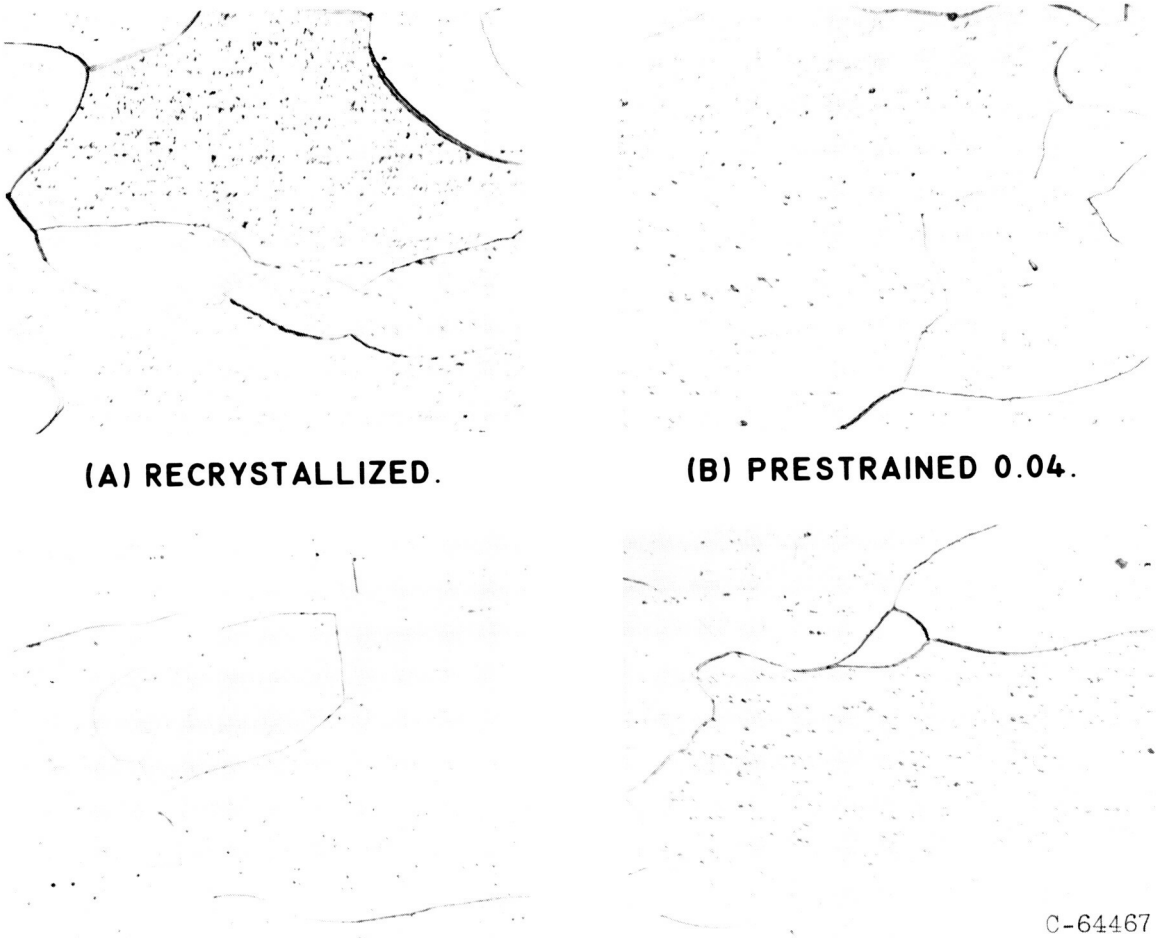

Figure 8. - Microstructure of 0.04 prestrained tungsten. Carbon content, 8 ppm. ×500.

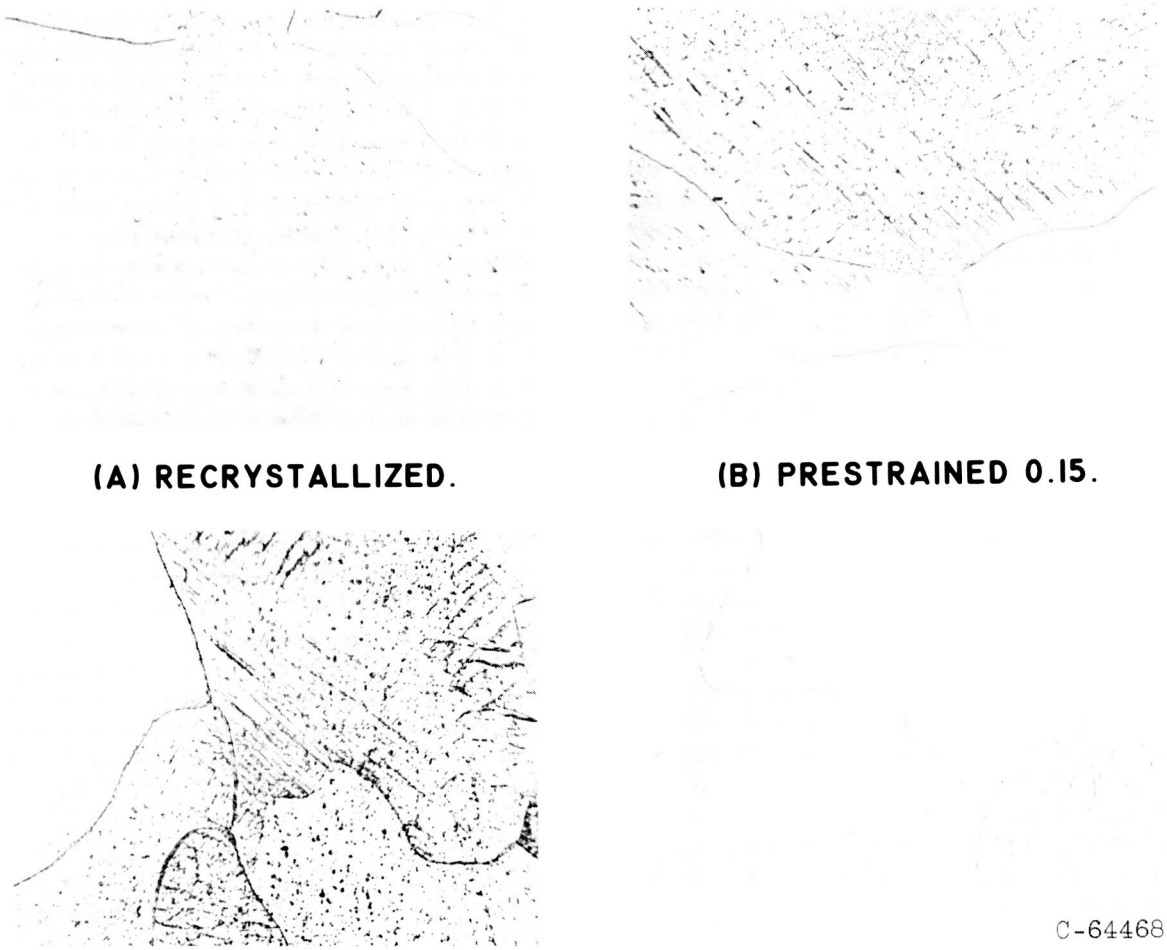

Figure 9. - Microstructure of 0.15 prestrained tungsten. Carbon content, 8 ppm. ×500.

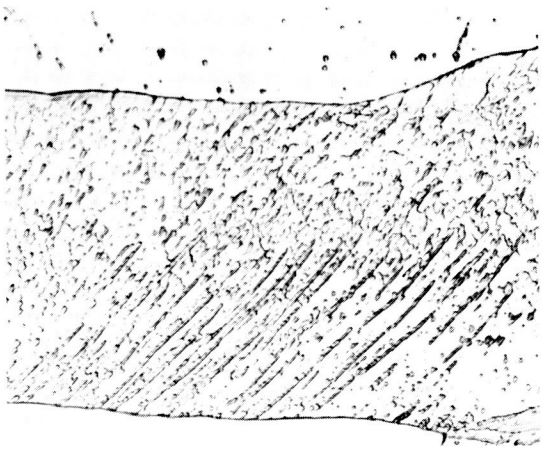

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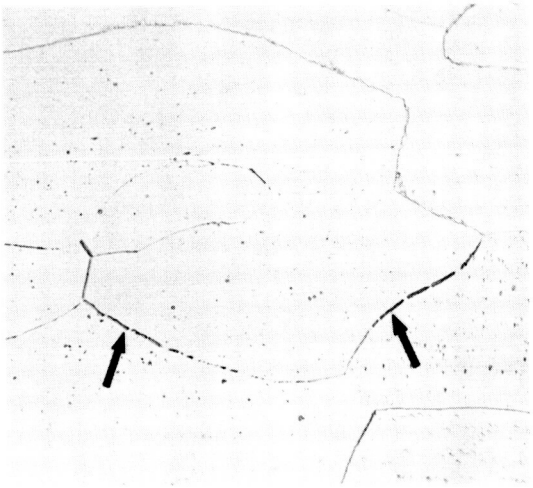
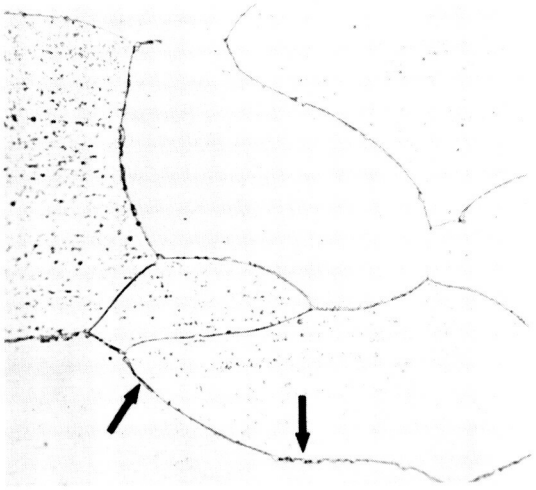
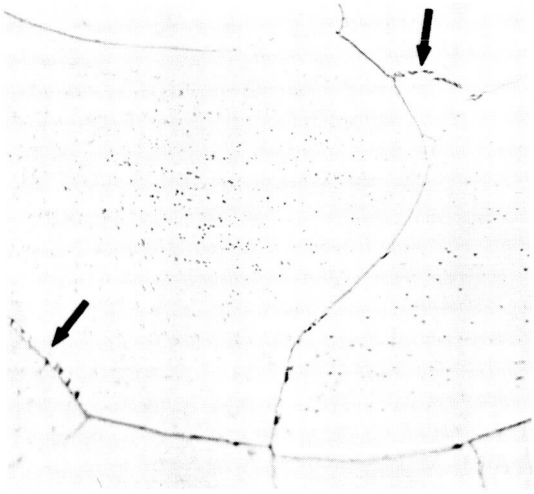
Figure 10. - Substructure of 0.15 prestrained tungsten. Carbon content, 8 ppm. ×1000.



(A) RECRYSTALLIZED. CARBON CONTENT, 40 PPM.



(B) PRESTRAINED 0.04.

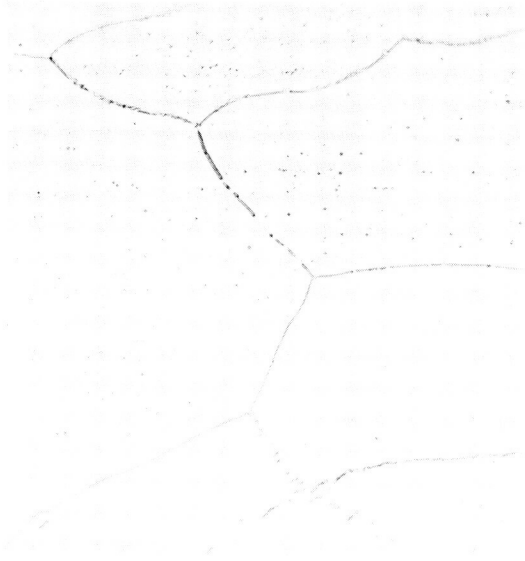


(C) AGED 30 MINUTES AT 1800° F.

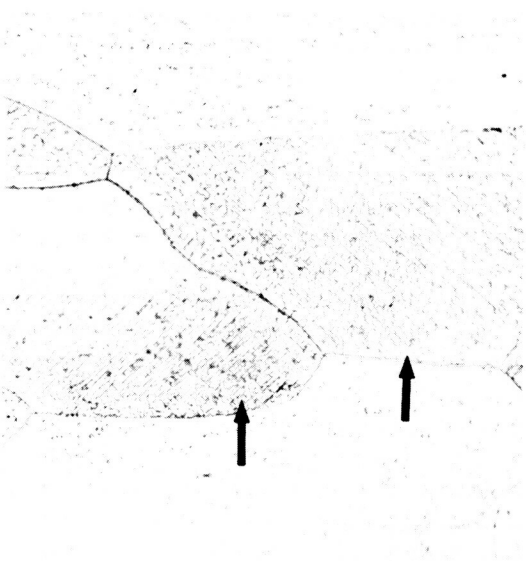
C-64463

(D) AGED 120 MINUTES AT 1800° F.

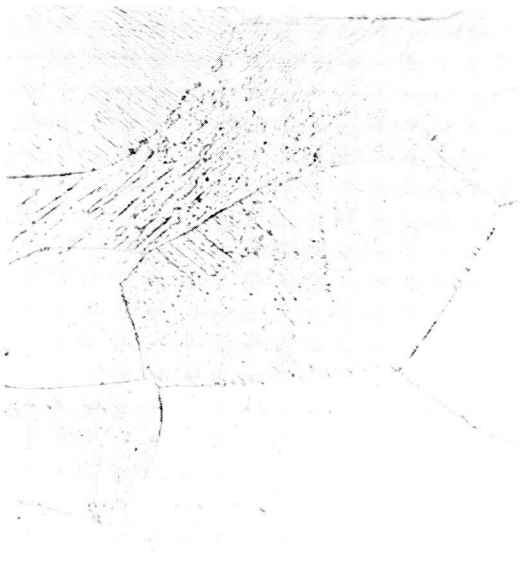
Figure 11. - Microstructures of 0.04 prestrained tungsten. Carbon content, 40 ppm. ×500.



(A) RECRYSTALLIZED. CARBON CONTENT, 40 PPM.



(B) PRESTRAINED 0.15.

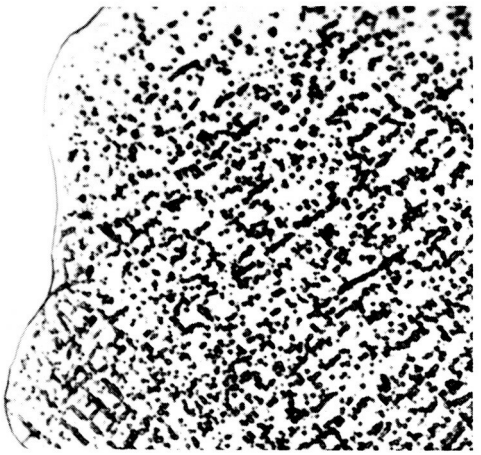


(C) AGED 30 MINUTES AT 1800° F.

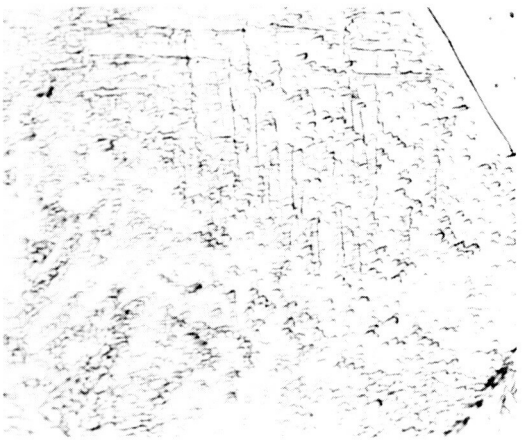
C-64465

(D) AGED 120 MINUTES AT 1800° F.

Figure 12. - Microstructures of 0.15 prestrained tungsten. Carbon content, 40 ppm. ×500.



(A) PRESTRAINED 0.15.



C-64466

(B) AGED 120 MINUTES AT 1800° F.

Figure 13. - Substructure of 0.15 prestrained tungsten. Carbon content, 40 ppm. ×1000.